

CITRUS

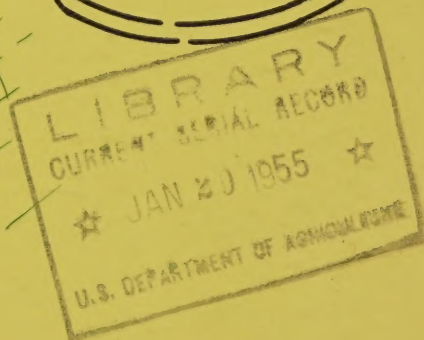
PROCESSING CONFERENCE

OCTOBER 7, 1954

Reserve

1,932

A4A68



UNITED STATES DEPARTMENT OF AGRICULTURE

Agricultural Research Service

Southern Utilization Research Branch

U. S. Citrus Products Station

Winter Haven, Florida

LH

LIST OF ATTENDANCE

Fourth Annual
CITRUS PROCESSING CONFERENCE
Florida Room, Citrus Building
Winter Haven, Florida
October 7, 1954

UNITED STATES DEPARTMENT OF AGRICULTURE
Agricultural Research Service
Southern Utilization Research Branch
U. S. Citrus Products Station

Altenburger, Michael, Breyer Ice Cream Company, Plant City, Fla.
Anderson, E. S., Food Machinery & Chemical Corp., Lakeland, Fla.
Barron, R. W., Florida Citrus Commission, Lake Alfred, Fla.
Beavens, E. A., Fruit & Vegetable Chemistry Laboratory, Pasadena, Calif.
Bennett, W. A., Citrus Experiment Station, Lake Alfred, Fla.
Bissett, Owen W., U. S. Citrus Products Station, Winter Haven, Fla.
Black, Joseph H., 951 Hollingsworth Road, Lakeland, Fla.
Bonnell, J. M., Clinton Foods Inc., Dunedin, Fla.
Bradner, Saidee E., Fosgate Citrus Concentrate Co-op., Orlando, Fla.
Brent, J. A., Minute Maid Corp., Plymouth, Fla.
Bristow, J. J., Clinton Foods Inc., Dunedin, Fla.
Boyd, John M., Continental Can Company, 4645 W. Grand, Chicago, Ill.
Brown, W. W., Minute Maid Corp., Plymouth, Fla.
Bunting, J. H., Campbell Soup Company, Camden, N. J.
Burg, Lilly E., Plymouth Citrus Products Co-op., Plymouth, Fla.
Byer, Ellis M., Florence Foods, Inc., Florence Villa, Fla.
Carter, R. D., Cantrell & Cochrane Peninsular Corp., Lake Alfred, Fla.
Chambers, Wm. C., Fosgate Citrus Concentrate Co-op., Orlando, Fla.
Childress, R. L., Marketing Economist, Federal Extension Service, U.S.D.A.,
Washington, D. C.
Chissom, G. A., Corn Products Sales Co., Leesburg, Fla.
Clark, C. B., Minute Maid Corp., Plymouth, Fla.
Colon, Luis Felip, Agr. Expt. Sta., Univ. of Porto Rico, Rio Piedras, P. R.
Conley, T. F., W. F. & John Barnes Company, 301 S. Water, Rockford, Ill.
Cook, R. W., Clinton Foods Inc., Dunedin, Fla.
D'Ercole, A. D., Birds-Eye Div., General Foods Corp., Box 481, Lakeland, Fla.
Deszyck, E. J., Citrus Experiment Station, Lake Alfred, Fla.
Dougherty, M. H., Florida Citrus Commission, Lake Alfred, Fla.
Dowling, Jack, Continental Can Company, 4645 W. Grand, Chicago, Ill.
DuBois, C. W., Minute Maid Corp., Plymouth, Fla.
English, Lamar, U.S.D.A. Processed Foods, Winter Haven, Fla.
Enns, Wm., Continental Can Company, Winter Haven, Fla.
Erwin, T. C., Atlantic Scientific Corp., Orlando, Fla.
Eskew, R. K., Eastern Regional Research Laboratory, Philadelphia, Pa.
Estabrooks, R. G., Minute Maid Corp., Leesburg, Fla.

Fehlberg, E. R., Pasco Packing Company, Dade City, Fla.
Fitzgerald, W. T., Minute Maid Corp., Plymouth, Fla.
Fowler, Wm., Continental Can Company, Winter Haven, Fla.
Garrison, Hugh, Birds-Eye Div., General Foods Inc., Box 481, Lakeland, Fla.
Gerwe, R. D., Food Machinery & Chemical Corp., Lakeland, Fla.
Giddings, W. W., Polk Packing Assn., Winter Haven, Fla.
Gifford, A. D., Continental Can Company, 100 E. 42nd Street, New York 17, N.Y.
Grist, Jack D., U.S.D.A. Processed Foods, Winter Haven, Fla.
Guyer, R. B., Continental Can Company, 4645 W. Grand, Chicago, Ill.
Hamrick, David, Fruit Industries, Inc., Bradenton, Fla.
Hendrickson, R., Citrus Experiment Station, Lake Alfred, Fla.
Hendrix, C. M. Jr., Winter Garden Citrus Products Co-op., Winter Garden, Fla.
Hill, E. C., Florida Citrus Commission, Lake Alfred, Fla.
Hinson, W. H., Winter Garden Citrus Products Co-op., Winter Garden, Fla.
Hughes, P. L., B. T. Smith Concentrates, 526 E. Lime, Lakeland, Fla.
Huggart, Richard, Florida Citrus Commission, Lake Alfred, Fla.
Hutto, T. H., Winter Garden Citrus Products Co-op., Winter Garden, Fla.
Jimenez, W. A., Minute Maid Corp., Plymouth, Fla.
Johnson, Joe, Food Machinery & Chemical Corp., Box 1642, Orlando, Fla.
Jones, H. L., Brown Citrus Machinery Corp., Winter Haven, Fla.
Kaplow, Milton, University of Miami, Coral Gables, Fla.
Kendrick, Robert, Pasco Packing Co., Dade City, Fla.
Kesterson, J. W., Citrus Experiment Station, Lake Alfred, Fla.
Kew, Theo. J., U. S. Citrus Products Station, Winter Haven, Fla.
Kime, J. A., Southern Regional Research Laboratory, New Orleans, La.
Lewis, J. Milton, University of Miami, Coral Gables, Fla.
Lime, B. J., U. S. Fruit & Vegetable Products Laboratory, Weslaco, Texas
Lyle, Mary C., Bordo Products Company, Winter Haven, Fla.
Madsen, H. S., Birds-Eye Div., General Foods Corp., Box 481, Lakeland, Fla.
Marshall, J. R., American Can Company, Tampa, Fla.
Marston, Harold, Clinton Foods Inc., Dunedin, Fla.
McAllister, Joe, U.S.D.A. Processed Foods, Winter Haven, Fla.
McDuff, O. R., Adams Packing Assn., Auburndale, Fla.
McFarlane, V. H., Southern Regional Research Laboratory, New Orleans, La.
McKinnis, R. B., Brown Citrus Machinery Corp., Winter Haven, Fla.
McNary, R. R., Florida Citrus Commission, Lake Alfred, Fla.
Miller, Don, Pasco Packing Co., Dade City, Fla.
Miller, W. M., Continental Can Company, 4645 W. Grand, Chicago, Ill.
Mitchell, W. G., Pasco Packing Co., Dade City, Fla.
Moore, E. P., B & W Canning Co., Groveland, Fla.
Morgan, D. A., U. S. Citrus Products Station, Winter Haven, Fla.
Mullon, W. S., Suni Citrus Products Co., Box 67, Haines City, Fla.
Murdock, D. I., Minute Maid Corp., Plymouth, Fla.
Nanz, Robert, Florida Chemists & Engineers Inc., 1709 N. Mills, Orlando, Fla.
Newhall, W. F., Citrus Experiment Station, Lake Alfred, Fla.
O'Brien, D. J., American Can Company, Tampa, Fla.
Olander, John W., W. F. & John Barnes Co., 301 S. Water, Rockford, Ill.
Olsen, R. W., Citrus Experiment Station, Lake Alfred, Fla.

Parker, J. L., Winter Garden Citrus Products Co-op., Winter Garden, Fla.
Parry, Wally, W. F. & John Barnes Co., 301 S. Waters, Rockford, Ill.
Patton, W. C., Campbell Soup, Winter Haven, Fla.
Peck, J. S., Joe Peck Inc., Winter Haven, Fla.
Pierce, S. K. Jr., Minute Maid Corp., Plymouth Fla.
Poitras, F. J., Florida Citrus Products Exchange, Box 2349, Tampa, Fla.
Pond, W. D., U.S.D.A. Processed Foods, Winter Haven, Fla.
Praschan, V. C., Clinton Foods Inc., Auburndale, Fla.
Pulley, G. N., Polk Packing Assn., Winter Haven, Fla.
Ratcliff, M. W., Florida Citrus Cannery Co-op., Lake Wales, Fla.
Riester, D. W., American Can Company, Tampa, Fla.
Robinson, R. D., Dr. P. Phillips Canning Co., Orlando, Fla.
Rouse, A. H., Citrus Experiment Station, Lake Alfred, Fla.
Roy, W. R., Minute Maid Corp., Plymouth, Fla.
Rushing, N. B., U. S. Citrus Products Station, Winter Haven, Fla.
Sanches, F., Agri. Expt. Sta., Univ. of Puerto Rico, Rio Piedras, P. R.
Scott, W. C., U. S. Citrus Products Station, Winter Haven, Fla.
Senn, V. J., U. S. Citrus Products Station, Winter Haven, Fla.
Singleton, Gray, Wm. J. Horsey Corp, Plant City, Fla.
Smith, F. C., 1231 - 13th St., N.W., Winter Haven, Fla.
Soule, M. J. Jr., U.S.D.A. Agric. Marketing Service, Orlando, Fla.
Stahl, A. L., University of Miami, Coral Gables, Fla.
Sterling, H. O., Citrus Experiment Station, Lake Alfred, Fla.
Stivender, F. H., Food Machinery & Chemical Corp., Lakeland, Fla.
Swift, L. J., U. S. Citrus Products Station, Winter Haven, Fla.
Terrien, Don, Continental Can Company, Winter Haven, Fla.
Tharp, W. R., Citrus, Inc., Haines City, Fla.
Thompson, E. A., Plymouth Citrus Products Co-op., Plymouth, Fla.
Tidwell, J. D., Bordo Products Company, Winter Haven, Fla.
Ting, S. V., Florida Citrus Commission, Lake Alfred, Fla.
Todd, Howard, Florida Citrus Cannery Co-op., Lake Wales, Fla.
Troy, V. S., Continental Can Company, 4645 W. Grand, Chicago, Ill.
Truitt, George, Clinton Foods Inc., Auburndale, Fla.
Trumm, Howard, Libby, McNeill & Libby, Ocala, Fla.
Tuthill, Palmer, Florida Citrus Cannery Co-op., Lake Wales, Fla.
Veldhuis, M. K., U. S. Citrus Products Station, Winter Haven, Fla.
Walker, S. S., Thornton & Company, Box 2880, Tampa, Fla.
Warren, J. B., Plymouth Citrus Products Station, Plymouth, Fla.
Watson, R. P., Market Coverage Co., 201 Maynard St., Orlando, Fla.
Wenzel, F. W., Florida Citrus Experiment Station, Lake Alfred, Fla.
Wineker, J., Food Machinery & Chemical Corp., Lakeland, Fla.
Wolford, E. R., U. S. Fruit & Vegetable Chemistry Laboratory, Pasadena, Calif.
Wolford, R. W., Florida Citrus Commission, Lake Alfred, Florida

PROGRAM AND ABSTRACTS OF PAPERS

FOURTH CITRUS PROCESSING CONFERENCE

October 7, 1954

Florida Room, Citrus Building
Winter Haven, Florida

It would be appreciated if no part of any abstract in this Program and Abstracts of Papers is reproduced or used without consulting the author or organization concerned. The abstracts were prepared for the benefit of those attending the conference and are not available for general distribution.

FOREWORD

This is the fourth Citrus Processing Conference held by Southern Utilization Research Branch at its U. S. Citrus Products Station in Winter Haven. A fall date was selected, as in 1953, since a meeting at this time of year seems to meet with the general approval of the representatives of industry and research organizations who would normally attend. It is hoped that this arrangement will permit a maximum number of those interested to attend.

The need for research in the citrus industry continues. The present favorable position of the citrus industry is due in large measure to its support of research and the rapidity with which the laboratory results have been applied. It is not likely that the gains accruing to the citrus industry may be maintained without continued research. Therefore industry should be promptly informed of research developments in order that the results may continue to improve processing procedures and to increase utilization. A conference provides an effective means of presenting information and of promoting the cooperation which is so essential to progress. These thoughts have motivated the organization of the 1954 conference as well as those preceding it.

The 1954 conference, like those before it, has benefited from the cooperation of various organizations. The Southern Utilization Research Branch, through its field station, the U. S. Citrus Products Station, expresses appreciation to the U.S.D.A. Citrus Fruit Advisory Committee, the Continental Can Company, Chicago, Illinois, the Florida Canner's Association, the Florida Citrus Products Research Panel, and to all others who have participated in the development of the program and contributed to its success. Thanks are also due to the Florida State Department of Agriculture and to the Florida Club for the use of the Florida Room, Florida Citrus Building in Winter Haven as a meeting place.

The four laboratories of the Utilization Research, Agricultural Research Service, U. S. Department of Agriculture, participating in the conference are: Citrus Products Station, Winter Haven, Florida; Fruit and Vegetable Products Laboratory, Weslaco, Texas; Fruit and Vegetable Chemistry Laboratory, Pasadena, California; and Eastern Regional Research Laboratory, Philadelphia, Pennsylvania.

PROGRAM
of
CITRUS PROCESSING CONFERENCE
October 7, 1954

MORNING SESSION

Chairman: M. K. Veldhuis, In Charge
U. S. Citrus Products Station
Winter Haven, Florida

9:30 A.M. Opening Remarks

		<u>Page</u>
9:40	Proposed Uniform Method of Designating Cloud Density Values. Vincent J. Senn, U. S. Citrus Products Station, Winter Haven, Florida and Robert T. O'Connor, Southern Regional Research Laboratory, New Orleans, Louisiana.	1
10:00	Growth Rates of Leuconostoc and Lactobacilli in Single Strength Orange Juice at Various pH Levels. N. B. Rushing, U. S. Citrus Products Station.	2
10:20	Some Constituents of Orange Peel. Lyle J. Swift, U. S. Citrus Products Station.	4
10:40	Highlights of the Citrus Research Program at the U. S. Fruit and Vegetable Chemistry Laboratory, Pasadena, California. (No abstract) E. A. Beavens, In Charge.	
11:00	Volatile Flavoring Constituents of Orange Juice. J. G. Kirchner and John M. Miller, U. S. Fruit and Vegetable Chemistry Laboratory. (To be given by Dr. Beavens)	5
11:20	Powders from Non-Citrus Fruit Juices. Roderick K. Eskew, Eastern Regional Research Laboratory, Philadelphia, Pennsylvania.	6
12:00 Noon	Recess for lunch	

AFTERNOON SESSION

Chairman: Vernon H. McFarlane, Head
Fruit and Vegetable Section
Southern Regional Research Laboratory
New Orleans, Louisiana

	<u>Page</u>
1:30 P.M. Processing Characteristics of Colored Texas Grapefruit. I Color and Maturity Studies of Ruby Red Grapefruit. Bruce J. Lime, Thomas S. Stephens, and Francis P. Griffiths, U. S. Fruit and Vegetable Products Laboratory, Weslaco, Texas.	7
2:00 Volatile Flavoring Constituents of Grapefruit Juice. J. G. Kirchner and John M. Miller, U. S. Fruit and Vegetable Chemistry Laboratory. (To be given by Dr. Beavens)	8
2:20 Preliminary Report of Investigations of Enzyme Systems Which May be Associated With Off-Flavors. Vincent J. Senn, U. S. Citrus Products Station.	10
2:40 Orange Concentrate Storage Studies. Lyle J. Swift, U. S. Citrus Products Station.	11
3:00 A Comparison of Several Methods of Heat Stabilization of Florida Frozen Orange Concentrate ^{a/} Owen W. Bissett and M. K. Veldhuis, U. S. Citrus Products Station, and Richard B. Guyer and W. M. Miller, Continental Can Company, Incorporated, Chicago, Illinois.	12

^{a/} Report of progress of cooperative work conducted at the U. S. Citrus Products Station under a Memorandum of Understanding between the Continental Can Company, Incorporated, Chicago, Illinois, and the Southern Utilization Research Branch, Agricultural Research Service, U. S. Department of Agriculture, Washington, D. C.

PROPOSED UNIFORM METHOD OF DESIGNATING CLOUD DENSITY VALUES

VINCENT J. SENN
Citrus Products Station
Winter Haven, Florida

and

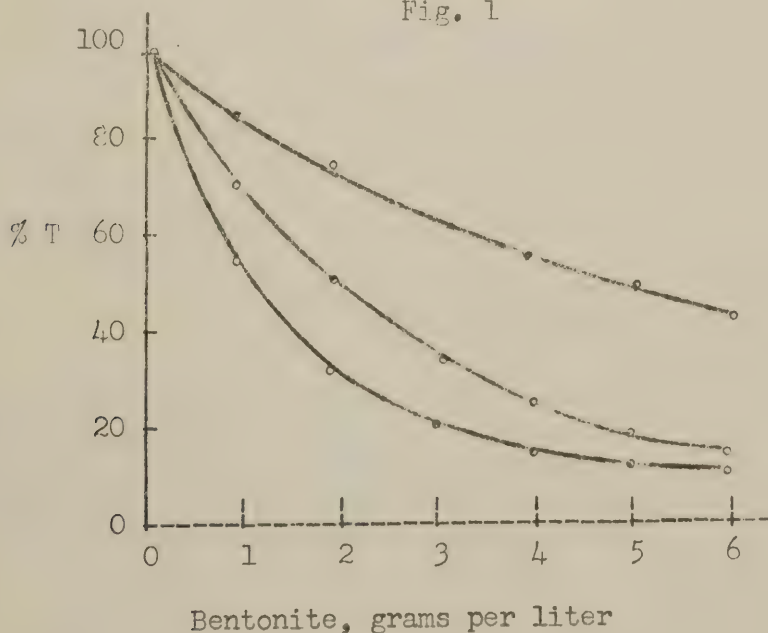
ROBERT T. O'CONNOR
Southern Regional Research Lab.
New Orleans, La.

Cloud density in citrus juices is reported in the literature as "per cent transmission" or as "galvanometer reading" in whatever units may be employed on the galvanometer or slide wire of the particular instrument. The variability between instruments of different brands and even between instruments of the same brand and model makes it impossible to obtain satisfactory precision in investigations involving cloud density values in many cases. This variability is illustrated for the popular Lumetron Model 401 in figure 1.

Suspensions of barium sulfate have been suggested as turbidity standards for citrus juices, but the well-known limitations of this compound as a turbidity standard have made it desirable that something better be sought. Bentonite suspensions appear to be suitable for this purpose. It is suggested that cloud density values be reported as the concentration in grams per liter of a standard bentonite. The use of such an artificial standard will make it possible to compare samples widely separated in space or time with suitable precision. Storage studies, for example, would be protected against changes in instrumentation.

It is suggested that such a standard would facilitate estimation of the stability of the product by permitting the use of a value such as the time required for the cloud density under any given set of conditions to reach some definite fraction of its initial value, such as one-half or one-third. This could be designated as the $t_{1/2}$ or $t_{1/3}$, simplifying recording or reporting.

Fig. 1



GROWTH RATES OF LEUCONOSTOC AND LACTOBACILLI IN SINGLE-STRENGTH ORANGE JUICE AT VARIOUS pH LEVELS

N. B. RUSHING
Citrus Products Station
Winter Haven, Florida

Considerable discussion has developed in the citrus industry concerning the growth of organisms producing "off-flavors" in orange juice prior to and during concentration. Consensus of opinions was that the pH of the juice was the determining factor. If the pH of the juice was below a certain level, generally conceded to be pH 3.8, then there was no "off-flavor" produced. These results are the first phase of a series of experiments to determine the growth rates of *Leuconostoc* and *Lactobacilli* in single-strength orange juice and concentrates adjusted to pH 3.4, 3.6, 3.8 and 4.0.

Two strains each of *Leuconostoc dextranicum*, *Leuconostoc mesenteroides*, *Lactobacillus brevis*, and *Lactobacillus plantarum*, var. *mobilis* were grown in single-strength orange juices adjusted to pH 3.4, 3.6, 3.8 and 4.0. Initial and hourly portions for seven hours were plated on orange serum agar. These inoculated orange juices were continuously shaken during the experiments. Growth rates were calculated from log 10 number of organisms per ml. using the data obtained from these plate counts. The growth rate is defined as the slope of the curve derived by plotting the logarithm of the number of organisms per ml. against time. This should give a curve of the type $y = a + bx$, where:

a = constant

n = number of platings

x = time in hours

b = growth rate

y = log 10 number organisms per ml.

The value of b was calculated from the relationship:

$$b = \frac{\sum xy - \frac{\sum x \sum y}{n}}{\sum x^2 - \frac{(\sum x)^2}{n}}$$

Some variation was observed between the three runs of the same organism at the same pH. Increase in pH from 3.4 to 4.0 caused a corresponding increase in growth rate with *Leuconostoc* more easily influenced than *Lactobacilli*. The growth rates obtained for these bacteria in single-strength (12° Brix) orange juice are tabulated in the following table:

Average growth rates

Organism	No. of runs	pH 3.4	pH 3.6	pH 3.8	pH 4.0
<u>Lact. brevis</u>	ave. of 3	.031	.045	.077	.099
" " "	" " "	.043	.047	.076	.111
" " 6	" " 6	.037	.046	.077	.105
<u>Lact. plantarum,</u> <u>var. mobilis</u>	ave. of 3	.041	.055	.083	.110
" " "	" " "	.046	.063	.084	.113
" " 6	" " 6	.043	.059	.083	.111
<u>Leuco. dextranicum</u>	ave. of 2	.061	.090	.130	.192
" " 3	" " 3	.060	.105	.127	.180
" " 5	" " 5	.061	.097	.128	.186
<u>Leuco. mesenteroides</u>	ave. of 3	-.021	.029	.126	.176
" " "	" " "	-.013	.058	.148	.220
" " 6	" " 6	-.017	.044	.137	.198

Relative growth rates as %

<u>Lact. brevis</u>	100%	123%	206%	280%
<u>L. plantarum, var. mobilis</u>	100%	135%	191%	255%
<u>Leuco. dextranicum</u>	100%	162%	210%	303%
<u>Leuco. mesenteroides</u>	negative	-	-	-
<u>Lact. brevis</u>	81%	100%	167%	228%
<u>L. plantarum, var. mobilis</u>	74%	100%	141%	188%
<u>Leuco. dextranicum</u>	62%	100%	131%	189%
<u>Leuco. mesenteroides</u>	negative	100%	312%	451%

SOME CONSTITUENTS OF ORANGE PEEL

LYLE J. SWIFT
Citrus Products Station
Winter Haven, Florida

This work was undertaken to determine whether excessive press and finisher pressures incorporated any ill-flavored substances into citrus juices. The approach chosen to the problem was a chemical search among the constituents of peel, rag, and seed for the classes of compounds thought most likely to influence flavor. Among the possibilities considered were flavonoid compounds, alkaloids, tannic substances, and less well-characterized substances such as limonin and nomilin.

Work on a laboratory extract of early season orange peel yielded a flavonoid substance occurring in the free state (not as a glucoside). This appears to be a heptamethoxy flavone, but this judgment is based solely on the reactions and general chemical behavior and on the percentage of the methoxyl group. As of the date of this writing (September 20, 1954) the carbon and hydrogen data are not available for this compound or for those mentioned later.

Subsequent work was done on late season effluent from peel oil centrifugals. An isolation, similar to the one noted above, yielded a different freely-occurring flavonoid which fits the literature description for nobiletin, a hexamethoxyl flavone, previously isolated by Tseng from the Chinese drug chen-pi which, in turn, had been prepared from some variety of mandarin orange peel. The percentage of methoxyl group agrees well with theory for nobiletin, the reactions are in agreement, and certain of the degradation products have been tentatively identified and are compatible with this compound. In addition to those degradation products obtained by others, there is a possibility that one of the acidic products may finally establish the structure of this substance. This possibility is supported by a neutral equivalent measurement and by the methoxyl percentage.

In the course of the isolation of nobiletin there was isolated a substance that behaves like a phlobaphene which, in turn, implies the presence of certain types of tannic substances. However, an analysis made for these substances showed the content to be very small. Another fraction contains a crystalline substance that has not yet been investigated.

VOLATILE FLAVORING CONSTITUENTS OF ORANGE JUICE

J. G. KIRCHNER AND JOHN M. MILLER
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Work on the isolation and identification of the volatile flavoring constituents from 2500 gallons each of fresh, freshly-canned, and canned-stored orange juice was recently completed.

Analysis of the water-soluble constituents indicated an increase in ethyl alcohol in the two canned juices, and a considerable increase in methyl alcohol in the stored juice. Acetic acid, although present in the fresh juice, was progressively higher in the freshly-canned and canned-stored juices. The same unsaturated acids found in the grapefruit juices were found in both lots of canned juice. The quantity of furfural was higher in the stored juice than in the freshly-canned juice, but it was less than that found in the stored grapefruit juice. Other minor constituents such as acetaldehyde, acetone, and hydrogen sulfide were found.

Twenty-nine compounds were identified in the oils of the three lots of orange juice making a total of forty-one compounds, including the water-soluble constituents. In the oil fractions the following compounds were found in all three juices: limonene, beta-myrcene, alpha-thujene (?), $C_{15}H_{24}$ (I), $C_{15}H_{24}$ (II), methyl alpha-ethyl n-caproate, citronellyl acetate, decanal, citroneal, $C_{15}H_{24}O$ (I), $C_{15}H_{22}O$, linalool, $C_{15}H_{26}O$ (I), carveol, decanol, octanol alpha-terpineol, hexanol, 3-hexen-1-ol, $C_7H_{16}O_2$, and polyoxygenated compounds. The following were found in the fresh and freshly canned juice, but not in the stored canned juice: ethyl isovalerate, ethyl $C_6H_8O_2$, terpinyl acetate, hexan-1-al, octan-1-al, n-2-dodecen-1-al (?), $C_{15}H_{24}O$ (II), and $C_{15}H_{26}O$ (II). Carvone was found only in the stored canned juice. The major changes which take place in the oil fraction of orange juice on canning and storage are: loss of total volatile oil, conversion of part of the hydrocarbons to alcohols, and loss of esters, aldehydes, and terpene aliphatic alcohols. Changes in the volatile oils have been evaluated for their flavor contribution to the canned and stored canned orange juices. The destruction of the more fruity ester, aldehyde, and alcohol portions of the oil, and the production of alpha-terpineol, carveol, and furfural imparts an off-flavor to orange juice, but the dominant off-flavor of canned and stored canned orange juice appears to arise from non-volatile precursors.

POWDERS FROM NON-CITRUS FRUIT JUICES

RODERICK K. ESKEW

Eastern Regional Research Laboratory
Philadelphia 18, Pennsylvania

A process is described for the preparation of powders from juices of non-citrus fruits. This procedure which was developed at the Eastern Utilization Research Branch is applicable to fruits whose aromas are recoverable in aqueous form, for example, Concord grapes and apples. The process differs from that used in making powdered citrus juices in that the aromas are restored prior to drying and by the addition of sugars and other substances are sufficiently retained during drying for the reconstituted product to possess a good flavor characteristic of the fruit. The powders store well for at least 6 months at 100° F.

PROCESSING CHARACTERISTICS OF COLORED TEXAS GRAPEFRUIT
I. Color and Maturity Studies of Ruby Red Grapefruit ^{1/}

BRUCE J. LIME, THOMAS S. STEPHENS, AND FRANCIS P. GRIFFITHS
U. S. Fruit and Vegetable Products Laboratory
Weslaco, Texas

In order to successfully process juice from colored grapefruit, more information was necessary about the development of color, and its relationship to maturity. For opening studies on this problem the Ruby Red was selected because it is probably the most widely planted of the colored varieties in Texas. Measurements of color, and some chemical and physical characteristics were made at intervals of two weeks from July, 1953 to April, 1954, during which time the fruit varied from very immature to definitely overmature. These measurements included visual and reflected color, average weight, Brix, acid, naringin, lycopene and carotene content.

During the period covered, the average weight per fruit increased approximately 2.5 fold, from 207 to 543 gs. Acidity, calculated as citric, decreased from 2.43 to 1.0 percent. The Brix decreased from 12.5° to 10.0°. The percentage of naringin decreased as the fruit matured, but reached a plateau of approximately 0.030 percent at about the time (February) the fruit was full grown.

The percentage of lycopene increased rapidly during August, then began a sharp decline which lasted until the middle of November. The residual amount decreased slightly from November to May. Carotene gradually increased during the growth of the fruit, from July through January; remained fairly constant until the middle of March, and then declined. From November 16 on the percentage of carotene exceeded the percentage of lycopene in the samples.

Visual color (redness) was judged "good" during July, August, and to the middle of September, and "excellent" during the latter part of September and through October. Color faded to a rating of "good" during November and part of December, and continued to fade to "fair" and "poor" as the season progressed. Reflected color measurements agreed with visual ratings after the fruit reached maximum coloration.

The data collected indicate that if processors desire maximum color consistent with good quality it will be necessary to process during a rather short period when naringin values are low, Brix-acid ratio is approximately 8, and while there is sufficient pigment in the pulp to give an attractive appearance.

^{1/} The complete manuscript is scheduled for publication in an early issue of Food Technology.

VOLATILE FLAVORING CONSTITUENTS OF GRAPEFRUIT JUICE

J. G. KIRCHNER AND JOHN M. MILLER
Fruit and Vegetable Chemistry Laboratory
Pasadena, California

Results of research on the isolation and identification of the volatile flavoring constituents distilled from 2500 gallons each of fresh, freshly-canned, and canned-stored grapefruit juice have been published.

There was little difference in the composition of the volatile water-soluble compounds of the fresh and freshly-canned juices. The latter contained a small amount of volatile acids, and a trace of furfural not found in the fresh juice. The canned-stored juice, however, had an increase in volatile acids, methyl alcohol, and furfural. The main acid found was acetic acid with traces of several unsaturated acids. Of these water-soluble compounds only the furfural contributed to off-flavors.

From the oil fractions of these juices, six hydrocarbons were isolated and identified. The main hydrocarbon, limonene, was found to play an important role in off-flavor development by conversion to alpha-terpineol and linalool monoxide during the storage period. The non-hydrocarbon volatile oil fraction is responsible for the typical fresh juice flavor. However, in the freshly-canned and canned-stored juices, this particular fraction contains the off-flavor components alpha-terpineol and linalool monoxide.

A total of thirty volatile compounds were found in the three juices examined as follows: Limonene, alpha-pinene, alpha-caryophyllene, beta-caryophyllene, a $C_{15}H_{24}$ hydrocarbon, a $C_{15}H_{28}$ hydrocarbon, N-methyl methylantranilate, caryophyllene oxide, citral, carvone, a nitrogen compound $C_{13}H_{15}N$, linalool, a compound $C_{15}H_{26}O$, carveol, a compound $C_{15}H_{22}O$, alpha-terpineol, 3-hexene-1-ol, geraniol, linalool monoxide, a compound $C_{12}H_{20}O_2$, acetaldehyde, acetone, furfural, ethyl alcohol, methyl alcohol, acetic acid, an acid $C_6H_8O_2$, a second acid of formula $C_6H_8O_2$, traces of an additional acid, and hydrogen sulfide.

Of the oil components geraniol, N-methyl methylantranilate, the $C_{15}H_{28}$ hydrocarbon, and the $C_{13}H_{15}N$ nitrogen compound were present only in the fresh and freshly-canned juices. The leaf alcohol 3-hexene-1-ol was found only in the fresh juice. There was only a small difference in the composition of the fresh and freshly-canned juices, but these differ greatly from the canned-stored juice. The most significant changes found were the decrease in limonene and a corresponding increase in alpha-terpineol and linalool monoxide. The latter two compounds and the furfural produced off-flavors when added to the fresh juice.

PUBLICATIONS

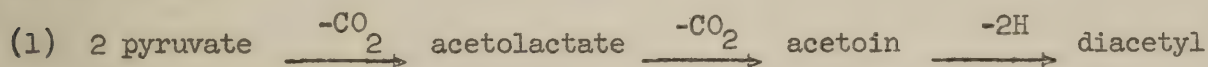
1. Separation and Identification of Terpenes by a New Chromatographic Technique. J. G. Kirchner, J. M. Miller, and G. J. Keller, Anal. Chem. 23 (3): 420-425, (1951).
2. Preparation of Terpeneless Essential Oils. A Chromatographic Process. J. G. Kirchner and J. M. Miller, Ind. and Eng. Chem. 44 (2): 318-21, (1952).
3. Some Improvements in Chromatographic Techniques for Terpenes. J. M. Miller and J. G. Kirchner, Anal. Chem. 24 (9): 1480-82, (1952).
4. Volatile Water-Soluble Constituents of Grapefruit Juice. J. G. Kirchner, J. M. Miller, R. G. Rice, G. J. Keller, and M. M. Fox, J. Agr. and Food Chem. 1 (7): 510-12, (1953).
5. Volatile Oil Constituents of Grapefruit Juice. J. G. Kirchner and J. M. Miller, J. Agr. and Food Chem. 1 (7): 512-18, (1953).
6. Chromatostrips for Identifying Constituents of Essential Oils. J. M. Miller and J. G. Kirchner, Anal. Chem. 25, 1107, (1953).

PRELIMINARY REPORT OF INVESTIGATIONS OF ENZYME SYSTEMS WHICH MAY
BE ASSOCIATED WITH OFF-FLAVORS

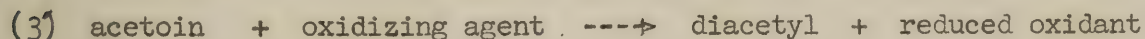
VINCENT J. SENN
Citrus Products Station
Winter Haven, Florida

The identification of diacetyl as the component responsible for "buttermilk off-flavor" in frozen orange concentrate made it desirable to determine the mechanism of formation of this compound. That buttermilk flavor can be produced by bacteria is not disputed, but it is possible that diacetyl may arise without active bacterial mediation. Voges-Proskauer active substances occur in orange juice under conditions which make their production by bacteria improbable. At least one of these substances may be acetylmethyl carbinol, also known as acetoin and as 3-hydroxy-butanone-2. At least this one V-P active compound can give rise to diacetyl.

It has been well established that acetoin arises by condensation and decarboxylation of two molecules of pyruvic acid. The enzyme responsible for this condensation has been named "carboligase." Most of the reported studies of carboligase activity refer to micro-organisms, but it has also been reported in higher plants, and its occurrence in orange fruits is a distinct possibility. One enzymatic mechanism proposed for the synthesis of diacetyl from pyruvate is as follows:



Alternatively, the acetoin may give rise to diacetyl either by dismutation of the acetoin (2) or by chemical oxidation (3) by an unidentified oxidizing agent.



Initial studies involved attempts to detect enzymatic dehydration (oxidation) of acetoin by the Thunberg technique. These studies indicated that any measurable reducing activity toward methylene blue is associated with the insoluble matter of the juice, and that reducing activity not associated with acetoin is sufficiently strong to mask any acetoin dehydrogenase activity. Attempts to demonstrate this last step in scheme (1) have been discontinued at this time.

It should be possible to study the first two steps of this scheme by the use of the Warburg microrespirometer to measure the rate of carbon dioxide production. Results are inconclusive as yet, but studies are continuing.

ORANGE CONCENTRATE STORAGE STUDIES

LYLE JAMES SWIFT
Citrus Products Station
Winter Haven, Florida

Storage studies were carried out on whole orange juice, filtered orange juice, and filtered juice with part of the cations removed. Each of these juices were investigated at 1-, 4-, and 6-fold concentration, with and without addition of limonene and terpeneless orange oil to the concentrates, and at storage temperatures of 40° and 80° F. Examinations of each pack at both temperatures were carried out at 1, 2, 3, 4, 7, 10 and 15-month storage periods. The examinations included observations on headspace pressure, total soluble solids (refractometer), pH, titratable acidity, color density (aqueous phase), ascorbic acid content and flavor.

Removal of the suspended matter from orange juice by filtration took away much of the characteristic flavor and did not markedly delay the deterioration as measured in the experiment.

At room temperatures or above, any flavor effect of peel oil or its constituents was quickly obscured by the profound deterioration changes arising from other causes. At refrigerator temperatures, the addition of limonene showed little effect on any of the measurements made, but the addition of terpeneless oil resulted almost immediately in an apricot-like flavor that persisted for the duration of the storage period.

Partial removal of cations from the filtered juice had little effect on the storage life.

Gas production, as indicated by headspace pressure measurements, showed a steady increase throughout the duration of the experiment at 80° F. This was almost true for color production in the aqueous phase. At 40° F. the changes were negligible.

Titratable acidity decreased somewhat with storage at 80° F. in all samples and there was a concomitant rise in pH. As far as the writer is aware, this phenomenon had not been previously observed.

The data on ascorbic acid showed that it was quite stable at refrigerator temperatures, but that at 80° F. there was a fairly rapid decomposition during the first few months. Up to this point the data agreed well with that of other investigators. However, after the first few months decline at the higher temperature, the ascorbic acid apparently increased. No such behavior has been reported by other workers in field, probably because most studies of this type have been terminated after shorter storage periods. A possible explanation for this phenomenon lies in the formation of reductones from sugar fragments or pectin degradation products such as uronic acids.

A COMPARISON OF SEVERAL METHODS OF HEAT STABILIZATION OF FLORIDA FROZEN ORANGE CONCENTRATE

R. B. GUYER AND W. M. MILLER
Research Division, Continental Can Company, Inc.

O. W. BISSETT AND M. K. VELDHUIS
Citrus Products Station
Winter Haven, Florida

During the 1952-53 and 1953-54 seasons a cooperative project was undertaken by the Research Division, Continental Can Company, Inc. and the U.S.D.A. at their Citrus Products Station in Winter Haven, Florida. The main objective of this project was to study the various aspects of heat stabilization of frozen orange concentrate. This investigation has included heat stabilization time and temperature studies, the effect of varietal and seasonal changes as related to heat stabilization, the effect of heat treatment at various stages of concentration on enzyme inactivation and cloud stability, a study of stability of high brix concentrate and a comparison of several methods of heat stabilization of frozen orange concentrate. It is with the latter study that this report is concerned.

The juice for the variables under study was extracted on a rotary juice press and was finished through a 0.033 inch screen. All of the juice for a day's run was extracted in that morning and stored in a refrigerated tank until used. The single-strength juice was heat stabilized in either a plate type heat exchanger, a small tube turbulent flow heat exchanger, or by direct steam injection at temperatures of 150° F., 160° F., 170° F. or 180° F. A Cherry Burrell ¹/₂ plate type heat exchanger equipped with a heating section and two cooling sections was used for the plate type variables. This unit was operated at 6 gallons per minute and the juice was at the desired temperature for approximately 3 to 5 seconds. A 1" - 1-1/2" Schutte and Koerting Company ¹/₂ stainless steel steam jet syphon was used for steam injection. The cooling sections of the plate type heat exchanger were used to cool the steam heated juice. The small tube turbulent flow exchanger consisted of 33 feet of 5/16 inch stainless steel tubing in a hot water jacket plus a 5 second hold tube and an annular cooler. The heat stabilized juice was concentrated in a falling film pilot plant evaporator to 55° Brix and was cut back to 42° Brix with unheated single-strength juice. It was then frozen in 202x214 cans in air blast at 0° F.

A summary of the results of the effect of the various heat treatments on enzyme activity and cloud stability are presented in Tables 1 and 2. The results show that steam injection does a more efficient job of reducing the enzyme activity at 150° F. than does a plate type or small tube heat exchanger at that temperature. At 160° F., 170° F. or 180° F. little difference was noted between the amount of pectinesterase activity remaining and the type of heat exchanger used. However, as shown in the results, the concentrate that was heat treated by steam injection remained stable for much longer periods when stored at 40° F. than the concentrate heat treated by either plate type or small tube turbulent flow heat exchanger which seems to indicate that direct steam injection has some as yet unidentified stabilizing effect other than the inactivation of the enzyme pectinesterase.

¹/₂ The mention of trade products does not imply that they are endorsed over similar products not mentioned.

Table 1 - The cloud stability of frozen orange concentrate made from Seedling fruit heat stabilized by a small tube turbulent flow heat exchanger and direct steam injection. Variables packed 3/54, Brix 14.7°, Acid 1.09%.

	Small Tube		Steam Injection	
	Days stable at 40° F.	Percent PE activity remaining	Days stable at 40° F.	Percent PE activity remaining
150° F.	14	56%	21	39%
160° F.	22	19%	35	17%
170° F.	21	20%	35	21%
180° F.	22	17%	42	19%

Table 2 - The cloud stability of frozen orange concentrate made from Valencia fruit heat stabilized by a small tube turbulent flow heat exchanger, by a plate type exchanger and by direct steam injection. Variables packed 4/54, Brix 11.3°, Acid 0.87%.

	Small Tube		Plate Type		Steam Injection	
	Days stable at 40°F.	Percent PE activity remaining	Days stable at 40°F.	Percent PE activity remaining	Days stable at 40°F.	Percent PE activity remaining
150° F.	6	61%	8	87%	12	41%
160° F.	14	26%	21	27%	26	28%
170° F.	12	19%	21	19%	35	22%
180° F.	23	13%	23	14%	42	19%

